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(54) Title of the Invention: Polypropylene Resin Sheet Manufacturing Method and Resin Sheet Obtained Thereby

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(72) Inventor: Atsushi FUJII
c/o Idemitsu Petrochemical Co., Ltd.
841-3 Kou, Shirahama-cho
Himeji-shi, Hyogo-ken

(72) Inventor: Kazuyuki FUKUDA
c/o Idemitsu Petrochemical Co., Ltd.
841-3 Kou, Shirahama-cho
Himeji-shi, Hyogo-ken

(71) Applicant: Idemitsu Petrochemical Co., Ltd.
1-1 Marunouchi 3-chome
Chuo-ku, Tokyo-to

(74) Agent: Tetsuo HOTAKA, Attorney

Specification Document

1. Title of the Invention

Polypropylene Resin Sheet Manufacturing Method and Resin Sheet Obtained Thereby

2. Scope of the Patent Claims

1. A manufacturing method for polypropylene type resin sheet comprising the steps of:
melting at a temperature greater than or equal to 200°C a resin composition formed by
blending an aliphatic amide type lubricant in the polypropylene type resin, extruding this
composition as a sheet, and thereafter rapidly cooling the sheet to a temperature less than
or equal to 30°C.
2. A manufacturing method for polypropylene type resin sheet comprising the steps of:
melting at a temperature greater than or equal to 200°C a resin composition formed by
blending an aliphatic amide type lubricant and a petroleum resin in the polypropylene
type resin, extruding this composition as a sheet, and thereafter rapidly cooling the sheet
to a temperature less than or equal to 30°C.

3. The manufacturing method for polypropylene type resin sheet according to claim 1 or claim 2, wherein the aliphatic amide type lubricant is an alkylene bis(amide).
4. The manufacturing method for polypropylene type resin sheet according to claim 1 or claim 2, wherein the rapid cooling is performed by introduction into a slit of downward falling cooling water at a temperature less than or equal to 20°C
5. A manufacturing method for polypropylene type resin sheet, wherein the sheet obtained by the method mentioned in any one of claims 1 through 4 is further subjected to heat treatment and/or elongation at 80°C to 160°C.
6. A polypropylene type resin sheet containing an aliphatic amide type lubricant and having less than or equal to 10% haze at 300 μ thickness.

3. Detailed Description of the Invention

[Industrial Field of Use]

This invention relates to a manufacturing method for a polypropylene type resin sheet that is capable of being used for molding of various types of wrapping containers of food products, medical products, miscellaneous goods, and the like, or being used as roll stock for elongated film, and this invention relates to the polypropylene type resin film that is obtained by this method.

[Conventional Technology]

In recent years, polypropylene type resin has been used for packaging of food products, medical products, miscellaneous goods, and the like as a film, sheet, or thermally molded container since this polypropylene type resin has good strength, heat resistance, moisture resistance, stiffness, and the like.

However, due to crystallinity of polypropylene, the sheet, film, and molded articles of polypropylene are insufficiently transparent. Methods that have been proposed to solve this problem include adoption of rapid cooling by rapidly cooling the extruded sheet, addition of a nucleation agent, addition of a petroleum resin, and combinations of such methods. Among these methods, although addition of petroleum resin and addition of nucleation agent are effective, the former method lowers resistance to mechanical shock, and solvent leaching resistance is insufficient. The latter method has the deficiencies of odor and insufficient resistance to solvent leaching.

That is to say, although a transparent polypropylene film is known that contains added 1,3,2,4-dibenzyliden-D-sorbitol and alkylene bis(amide) (nucleation agents) (Publication of Examined Patent Application No. S60-43859), in order to make the polyethylene film transparent using the nucleation agent as a main component, the utilized concentration of nucleation agent increases. However, this causes bleeding of the nucleation agent, resulting in odor and hot water leachability. The level of transparency is insufficient, and such problems make use of the polypropylene film for food product packaging applications difficult.

Moreover, a method is known (Publication of Unexamined Patent Application No. S58-134711) that obtains a glossy film by uni-directional elongation of a sheet containing a

bis(amide) compound blended at high concentration (0.5 - 5 PHR) in polypropylene resin. However, gloss and transparency in the sheet form are insufficient.

Moreover, it is known that it is possible to obtain a film that has excellent transparency and blocking by the use of the water-cooled blown film method to produce a film from a composition containing low density linear polyethylene, aliphatic amide, and a silicate added to a polypropylene copolymer (Publication of Unexamined Patent Application No. S62-246947), this method is effective for films less than or equal to 100 μ , but this method is insufficient for film sheets greater than or equal to 100 μ .

[Problem to be Solved by the Invention]

This invention was accomplished based on the above mentioned circumstances, and thus the problem is to utilize the characteristics of polypropylene resin by providing a method for obtaining a propylene type resin sheet that has excellent thermal molding properties during secondary processing of the sheet and that also has excellent transparency, stiffness, mechanical shock resistance, and solvent leaching resistance, while making possible manufacture of a polypropylene sheet that has a wide molding temperature range, has little draw-down, and has homogeneous thickness, thereby providing a sheet that can be used for numerous applications (firstly, food products) with neither odor nor bleeding.

Moreover, this invention does not limit the melt index of the base polypropylene, and this invention provides a method for manufacture of polypropylene type resin sheet that has excellent thickness, high speed molding performance, and transparency.

(Means to Solve the Problem)

As a result of an accumulation of dedicated research by the inventors of this invention in order to solve the above mentioned problem, this invention was achieved by discovery that a polypropylene resin sheet could be obtained that has excellent transparency and molding performance by molding under specific molding conditions a resin composition formed by blending specific additives with polypropylene type resin.

That is to say, this invention relates to a manufacturing method for a polypropylene type resin sheet comprising the steps of: melting at a temperature greater than or equal to 200°C a resin composition formed by blending an aliphatic amide type lubricant in the polypropylene type resin, extruding this composition as a sheet, and thereafter rapidly cooling the sheet to a temperature less than or equal to 30°C.

Homo-polypropylene, random polypropylene, block polypropylene, or a mixture of homo-polypropylene and random polypropylene can be used as the polypropylene type resin for this invention. A mixture can be used with advantage that contains at least 50 wt% homo-polypropylene. Normal crystalline polypropylene is used as this homo-polypropylene. Moreover, a copolymer of propylene and less than 20 wt% (preferably less than 15 wt%) of an α -olefin can be used as the random polypropylene. α -olefins that have a carbon number of 2 or a carbon number of 4 - 10 can be used with advantage as the α -olefin, wherein such compounds may be linear or branched. Specific examples that can be cited are ethylene, 1-butene, isobutene, 1-pentene, 3-methyl-1-butene, 1-hexene, 4-methyl-1-pentene, neohexene, 1-heptene, 1-octene, 1-decene, and the like, and ethylene and 1-butene and the like are preferred among these examples.

Furthermore, the above mentioned random polypropylene may be a copolymer of polypropylene and one type of the above mentioned α -olefins, or alternatively, may be a

copolymer of polypropylene and two or more types of the above mentioned α -olefins, or such copolymers may be used together.

The utilized polyolefin type resin can have a melt index (MI) value that is normally 0.5 - 20 g/10 min. and preferably is 0.5 - 5 g/10 min.

Although no particular limitation is placed on the aliphatic amide type lubricant used for this invention, specific examples of such aliphatic amide type lubricants are: stearyl amide (melting point of 100°C to 105°C), palmityl amide (melting point of 95°C to 100°C), oleyl amide (melting point of 68°C to 74°C), methylene bis(stearamide) (melting point of 138°C to 140°C), ethylene bis(stearamide) (melting point of 142°C to 145°C), and the like. The melting point is preferably greater than or equal to 100°C; and methylene bis(stearamide), ethylene bis(stearamide), and the like alkylene bis(amide)s are preferably used. When this aliphatic amide type lubricant is used, lubrication of the die face of the extruder die improves, it becomes possible to stably extrude a sheet that has a slick surface, and it is thought that transparency and surface gloss become good by this means.

Although no particular limitation is placed on the added concentration of aliphatic amide type lubricant, the added concentration is normally 0.02 - 2.0 wt% (polypropylene type resin composition basis) and is preferably 0.1 - 0.7 wt%. When this is less than 0.02 wt%, there is no transparency improvement effect. A concentration in excess of 2.0 wt% is undesirable due to the occurrence of problems such as discoloration, generation of smoke during manufacture, and the like.

Next, as may be required, it is possible to blend with the resin composition of this invention a low crystallinity or non-crystalline ethylene- α -olefin copolymer and/or petroleum resin within a concentration range of 1 - 20 wt%.

Examples of ethylene- α -olefin copolymers that can be cited for use for this invention are low crystallinity or non-crystalline copolymers obtained by copolymerization between ethylene and α -olefins such as propylene, butene-1, pentene-1, 3-methylbutene-1, 4-methylpentene-1, and the like. The utilized resin preferably has a density of 0.85 - 0.90 g/cm³.

Although no particular limitation is placed on the petroleum resin for this invention, the utilized petroleum resin is preferably an aliphatic type petroleum resin, cycloaliphatic type petroleum resin, or hydrogenated terpene type resin, or is a mixture of two or more types selected from among these resins. Transparency becomes improved due to blending of the petroleum resin.

It is possible to use known resins for this aliphatic type of petroleum resin, cycloaliphatic type petroleum resin, hydrogenated terpene type resin, and a mixture of such resins. Resins that can be used suitably have a molecular weight that is normally 400 - 5000, the softening point is preferably 50° to 160°C, and the degree of hydrogenation is preferably greater than or equal to 80%. Specific examples of the aliphatic type petroleum resin are olefins obtained by thermal decomposition of petroleum and the hydrogenation product of a hydrocarbon resin from diene type hydrocarbon polymerization (degree of polymerization = 5 - 20). Specific examples of the cycloaliphatic petroleum resin are the hydrogenation product of an aromatic type petroleum resin obtained by polymerization of a monomer (e.g., vinyl toluene, vinyl xylene, propenylbenzene, styrene, α -methylstyrene, indene, methylindene, ethylindene, and the like). Specific examples of the terpene type petroleum resin are polymers, copolymers, or hydrogenation products of such polymers from polymerization of cuts or fractions or acyclic, monocyclic or bicyclic mono-terpenes of various monomers such as allo-ocimene, carene, pinene, and the isomers thereof,

dipentene terpinene, terpinolene, limonene, and the like. Among the hydrogenated terpene type petroleum resins prepared by hydrogenation, preferred examples are those having a degree of hydrogenation greater than or equal to 80% and more preferably greater than or equal to 90%, although it is possible to use those in which unsaturated bonds and aromatic rings partially remain.

Furthermore, the above petroleum resin may be used individually or as a combination of two or more such petroleum resins.

Moreover, a small concentration of nucleation agent, inorganic fine powder, and the like may be added within limits not prejudicial to the object of this invention, preferably in an amount of 0.01 to 1 wt%. A known nucleation agent (organic or inorganic) may be used in this invention. Examples of inorganic powders that can be cited include silica, zeolite, hydrotalcite, and the like.

Furthermore, these compounds may be used individually or as a combination of two or more such compounds.

The manufacturing method of the polypropylene type resin sheet of this invention is characterized by manufacture of the polypropylene resin sheet by steps comprising: melting at a temperature greater than or equal to 200°C (preferably 210°C to 300°C) the above mentioned resin composition, extruding this composition as a sheet, and thereafter rapidly cooling the sheet to a temperature less than or equal to 30°C (preferably less than or equal to 20°C).

No particular limitation is placed on the order of blending of the melt kneading of the above mentioned various components, and these compounds may be added separately or simultaneously.

It is possible to perform this melt kneading beforehand separately by using a conventional melt kneading machine, but melt kneading can be suitably performed by using a melt extruder that has a melt kneading part. After performance of melt kneading, the molten polypropylene type resin composition is extruded through the die of a melt extruder to form a sheet.

Furthermore, as may be desired, normally utilized additives may be added to the above mentioned polypropylene type resin composition. Examples include stabilizers (e.g., thermal stabilizers, ultraviolet absorbers, anti-oxidants, and the like), lubricants, antistatic agents (e.g., various surfactants and the like), anti-fogging agents, and the like.

This melt kneading is performed using the melt extruder or the like, while maintaining the temperature of the resin composition at a temperature greater than or equal to 200°C and preferably a temperature within the range of 210°C to 300°C. If the temperature of melt kneading is less than 200°C, non-melted material will remain, kneading will become insufficient, and also extrusion power will become high. If the temperature of heating is excessively high, decomposition of the resin will occur, and cooling efficiency will drop.

Melt extrusion is normally performed using the T-die method. This is preferably carried out while the temperature for melting (kneading) the resin composition is maintained in the above mentioned temperature range. The die exit temperature is preferably a little higher than that of the above molten resin composition. For example, melt extrusion is normally performed while raising the die exit temperature */missing text/* 10°C to 60°C higher than the melt resin composition temperature. In this manner, due to setting of the temperature of the exit of the die somewhat higher than that of the molten resin composition, it is possible to maintain the state of the surface of the extruded sheet extremely well. Thereafter, by rapid cooling of this sheet to a temperature of less than or equal to 30°C and preferably less than or equal to 20°C, it is possible to provide good transparency. The lower this temperature, the better the transparency. The rapid

cooling is preferably performed by bringing the sheet into contact with cooling water that has a temperature less than or equal to 20°C. Rapid cooling is preferably performed by a slit-water cooling technique by use of cooling water that has a temperature less than or equal to 10°C.

This cooling water may be water alone, or alternatively, may be an aqueous solution obtained by adding an organic or inorganic thickening agent. The cooling water preferably has an added thickening agent for uniform cooling and surface smoothness. Here, examples of the utilized organic thickener are natural high molecular weight substances, semi-synthetic products, synthetic products, and the like. Examples of the natural high molecular weight substance include: starches (e.g., sweet potato starch, potato starch, wheat starch, and the like), mannans (e.g., such as that of alimentary yam paste and the like), seaweed-derived viscous substances (e.g., agar, sodium alginate, and the like), plant-derived viscous substances (e.g., tragacanth gum, gum arabic, and the like), microorganism-derived viscous substances (e.g., dextran, levan, and the like), proteins (e.g., glue, gelatin, casein, collagen, and the like). Examples of the utilized semi-synthetic product are: cellulose-based substances (e.g., viscose, methyl cellulose, carboxymethyl cellulose, and the like) and starch-derived substances (e.g., soluble starch, carboxymethyl starch, dialdehyde starch, and the like), and the like. Moreover, examples of the utilized synthetic product are polyvinyl alcohol, poly(sodium acrylate), polyethylene oxide, and the like.

Examples of the inorganic thickening agent are silica sol, alumina sol, clay, water glass, various types of metal salts, and the like.

In addition to the aqueous solutions prepared by adding these thickening agents to water, it is also possible to use a viscous substance alone such as polyethylene glycol, polypropylene glycol, silicone oil, and the like.

Viscosity of the aqueous solution containing the added thickening agent is normally 2 to 3000 centipoise (cP) and preferably is 3 to 1000 cP.

Here the utilized cooling device may, for example, be that disclosed in the Publication of Unexamined Patent Application No. S60-236719, that shown in Figure 1 of Publication of Unexamined Patent Application No. S61-130018, or a below mentioned cooling device shown in Figure 1 (downward-flowing cooling water type / water level controlled type cooling device).

An example of the rapid cooling procedure using the above mentioned water cooling method used by the method of this invention will be explained in further detail below while referring to the below mentioned Figure 1.

Figure 1 is a cross-sectional view conceptually showing an example of a cooling device used for the method of this invention. Within the figure, items 1 and 2 each indicate a water tank used for cooling water and water downward flow feed, respectively. Items 3 and 4 each represent a downward-flowing cooling water slit. Item 5 indicates a cooling water tank. Moreover, a resin composition sheet 9 extruded in the molten state from an extrusion T-die 6 is guided by cooled guide rolls 10 - 13 while downward flowing cooling water 7 and 8 are fed in the downward flow direction, and the resin composition sheet is thus guided into a cooling water 14 within the cooling water tank 5. After cooling is completed, the resin composition sheet is collected. The cooling water within the cooling water tank 5 can be temperature-controlled at a certain temperature by a suitable temperature control mechanism 15 and the like, and this cooling water can be recycled to the cooling water downward flow tanks 1 and 2 for reuse by a suitable pump 15. /TRANSLATOR'S NOTE: Item 16 is the pump, not item 15. This is an error in the source text./

Furthermore, the utilized cooling water downward flow slits are preferably the double type shown in Figure 1, and porous baffles are preferably used in the flow path within the water tanks. Furthermore, the downward flow velocity of the cooling water is particularly preferably faster than the feed velocity of the sheet so that cooling water does not pile up at the slit entrance port. In order to prevent this piling up, the water level within the water tank is as low as possible, preferably less than or equal to 7 mm.

Furthermore, no particular limitation is placed on the shape of the obtained polypropylene type resin composition sheet, and moldings can be formed as single layer sheets, multi-layer sheets, and the like that have the desired size (width) and (thickness). Based on the viewpoints of transparency, strength, and the like, thickness of this resin composition sheet is normally about 50 to 3000 μm and preferably is about 100 to 2000 μm . The method of this invention is effective from viewpoints such as the capability of maintaining high transparency even for a sheet that has a thickness particularly greater than or equal to 300 μm and the like. A polypropylene type resin sheet can be obtained that contains an aliphatic amide type lubricant and which has haze less than or equal to 10% at a thickness of 300 μm . Moreover, according to the method of this invention, not only is it possible to impart transparency to the obtained sheet, but it is also possible to markedly improve the surface state (i.e., gloss, appearance, and the like).

The resin sheet manufactured in this above mentioned manner may be subjected to heat treatment and/or elongation in order to improve transparency and stiffness.

As may be desired, the utilized heat treatment is carried out preferably in a temperature range of 60°C to 160°C and more preferably in a temperature range of 80°C to 150°C, and this treatment is preferably carried out for 1 second to 60 seconds and more preferably is carried out for 2 seconds to 30 seconds. This heat treatment may be carried out, for example, by use of a heating roll, heating air, inert liquids, and the like. Treatment is preferably performed by continuous treatment using a roll.

Moreover, during the above mentioned heat treatment or immediately after the heat treatment, elongation can be performed for improvement of thermal molding properties (e.g., drawdown prevention effect and the like).

This elongation is normally performed at 80°C to 160°C, preferably is performed at a temperature 5°C to 70°C lower than the melting point, and more preferably is performed at a temperature 5°C to 50°C lower than the melting point, and this elongation can be performed by roll elongation. Furthermore, the elongation can be conducted by uniaxial orientation or biaxial orientation. Elongation is preferably performed in the temperature range of 120°C to 150°C simultaneously with continuous heat treatment by rollers such that the width reduction rate is about 5 to 20%. No particular effect is obtained if elongation is carried out above this range, and reproducibility of molding at the time of thermoforming and the like are lowered due to elongation.

It is possible to obtain the desired polypropylene type resin sheet in the above mentioned manner.

The polypropylene type resin sheet obtained according to the method of this invention has superior properties such as strength, heat resistance, moisture permeation resistance, and the like, and also has the extremely excellent rigidity, mechanical shock resistance, and transparency of the original polypropylene resin without odor and the occurrence of bleeding. In the case of thermomolding, use is possible with advantage for a wide range of items (various types of packaging containers and the like) with excellent reproducibility, a wide range of molding

temperatures, and little thickness variation. Moreover, this sheet can be used for applications, in addition to thermomolded containers, such as case manufacturing by folding-bending processing and manufacture of roll stock for elongated film use (due to excellent elongation performance).

(Working Examples)

Although this invention is explained in detail below based on working examples, this invention is not limited to these examples.

(Working Examples 1 through 14, Comparative Examples 1 through 7)

A T-die extruder (extruder diameter = 90 mm, L/D = 24, die width = 850 mm, die lip opening = 2 mm) was used to extrude the polypropylene type resin compositions of the blended compositions indicated in Table 1 at a resin temperature of 230°C and a die temperature of 280° to melt-knead and extrude the compositions as transparent sheets. Thereafter the sheet was fed to a double slit type cooling water device as shown in Figure 1 (first stage slit: 50 mm tall, 2.5 mm wide, 4 mm water level, 5°C cooling water temperature, porous baffles used in the flow path, second state slit: 10 mm tall, 5 mm wide, 10 mm water level, 4°C cooling water temperature), and the sheet was rapidly cooled down to 4°C in 1 to 3 seconds to obtain a sheet of 0.3 mm thickness. This sheet was subjected to heat treatment using a 300 mm diameter roll (130°C). For certain sheets, elongation was performed at a width reduction rate of 10%. Results of physical property measurements, etc. of these sheets are shown in Table 1.

Codes within the table indicate the following.

H-PP1: IDEMITSU POLYPRO F200S (homo-polypropylene, MI = 2.0 g/10 min., produced by Idemitsu Petrochemical Co., Ltd.)

H-PP2: IDEMITSU POLYPRO E100GM (homo-polypropylene, MI = 0.5 g/10 min., produced by Idemitsu Petrochemical Co., Ltd.)

R-PP: IDEMITSU POLYPRO F744N (propylene-ethylene random copolymer, MI = 7.0 g/10 min., produced by Idemitsu Petrochemical Co., Ltd.)

PR: ARCON P-140 (cycloaliphatic petroleum resin, molecular weight = 1000, softening point = 140°C, produced by Arakawa Chemical Industries, Ltd.)

Lubrication agent: ethylene bis(stearylamide)

Nucleation agent: aluminum p-t-butyl benzoate

In addition, physical properties were measured in the below described manner.

Haze: JIS K-7105

Gloss: ASTM D-523

Tensile modulus: JIS K-7113

The air knife method of comparative examples 6 and 7 is a method for manufacture of a sheet by cooling-solidification using an air knife that presses air from a slit opposing a chill roll against the melt sheet resin leaving the die.

Table 1

	Resin blend				Heat treatment	Elongation	Thickness (mm)	Sheet physical properties		Tensile modulus (kg·cm / cm)	Comments
	wt. parts PP	wt. parts PR	lubrication agent (wt%)	nucleation agent (wt%)				haze (%)	gloss (%)		
working example 1	H-PP1 100		0.3		--		0.3	7	121	17,000/16,000	Air knife method. Roll temp. = 60°C.
working example 2	H-PP1 100		0.3		yes	--	0.3	5	121	19,000/19,000	
working example 3	H-PP1 100		0.3		yes	yes	0.3	4	122	21,500/21,000	
working example 4	H-PP1 100		0.3		yes	--	0.6	8	113	17,000/17,500	
working example 5	H-PP2 100		0.3		--	--	0.3	9	112	17,000/17,000	
working example 6	H-PP2 100		0.3		yes	--	0.6	12	104	18,500/18,500	
working example 7	R-PP 100		0.3		--	--	0.6	8	123	14,000/15,000	
working example 8	R-PP 100		0.3		yes	--	0.6	6	123	15,000/16,000	
working example 9	H-PP1 95	5	0.3		--	--	0.6	8	10	16,500/16,000	
working example 10	H-PP1 95	5	0.3		yes	--	0.6	8	111	17,500/18,000	
working example 11	H-PP1 95	5	0.3	0.15	--	--	0.6	6	120	18,000/18,500	
working example 12	H-PP1 95	5	0.3	0.15	yes	--	0.6	5	120	19,000/19,000	
working example 13	H-PP1 95	5	0.3	0.15	yes	yes	0.6	4	121	19,500/20,500	
working example 14	H-PP1 95	5	0.2	0.10	yes	--	0.6	6	118	18,000/19,000	
comparative example 1	H-PP1 100				--	--	0.3	12	106	17,000/16,500	
comparative example 2	H-PP1 100				yes	--	0.3	7	109	19,000/18,500	
comparative example 3	H-PP2 100				yes	--	0.6	18	100	17,500/17,000	
comparative example 4	H-PP2 100				yes	--	0.3	24	83	19,000/19,000	
comparative example 5	H-PP1 100				yes	--	0.6	36	81	16,500/17,000	
comparative example 6	H-PP1 100				--	--	0.3	26	air side = 74, roll side = 113	18,000/17,500	
comparative example 7	H-PP1 100				--	--	0.6	47	air side = 81, roll side = 112	16,000/17,000	

(Results of the Invention)

The sheet obtained according to this invention excellently utilizes the characteristics of polypropylene resin (heat resistance, moisture permeation resistance, and the like) without odor and generation of bleeding. The obtained sheet has excellent stiffness and gloss, extremely high transparency, excellent mechanical shock resistance and solvent leaching resistance, and excellent molding repeatability relative to vacuum molding conditions and the like. The sheet is capable of ready molding over a wide temperature range, and the sheet has the excellent characteristic of low drawdown.

4. Simple Description of the Figures

Figure 1 is a cross-sectional drawing showing conceptually one example of the cooling device used for the method of this invention.

Within the figure:

1 and 2	...	tanks used for cooling water downward flow feed
3 and 4	...	cooling water downward flow slits
5	...	cooling water tank
6	...	extrusion T-die
7 and 8	...	downward flow cooling water
9	...	resin sheet
10 - 13	...	guide rolls
14	...	cooling water
15	...	temperature adjustment mechanism
16	...	pump

Applicant: Idemitsu Petrochemical Co., Ltd.

Agent: Tetsuo HOTAKA, Attorney



Figure 1

